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FILE COVERS 1907 - 22 Aug 2004 VOL 141 ISS 9

FILE LAST UPDATED: 20 Aug 2004 (20040820/ED)

This file contains CAS Registry Numbers for easy and accurate substance identification.

=> s catalyst? (l) wax

851107 CATALYST?

73148 WAX

49346 WAXES

90570 WAX

(WAX OR WAXES)

L1 3574 CATALYST? (L) WAX.

=> s l1 and (regenerat? or rejuvenat? or activat? or reactivat?)

159352 REGENERAT?

2302 REJUVENAT?

1136027 ACTIVAT?

21751 REACTIVAT?

L2 250 L1 AND (REGENERAT? OR REJUVENAT? OR ACTIVAT? OR REACTIVAT?)

=> s l2 and (solvent or hexane or hexene or heptane or heptene or tetrahydrofuran)

608403 SOLVENT

302207 SOLVENTS

766333 SOLVENT

(SOLVENT OR SOLVENTS)

100535 HEXANE

1812 HEXANES

101544 HEXANE

(HEXANE OR HEXANES)

22066 HEXENE

1164 HEXENES

22581 HEXENE

(HEXENE OR HEXENES)

56183 HEPTANE

1160 HEPTANES

56785 HEPTANE

(HEPTANE OR HEPTANES)

6993 HEPTENE

577 HEPTENES

7312 HEPTENE

(HEPTENE OR HEPTENES)

37166 TETRAHYDROFURAN

1399 TETRAHYDROFURANS
 38136 TETRAHYDROFURAN
 (TETRAHYDROFURAN OR TETRAHYDROFURANS)
 96171 THF
 44 THFS
 96185 THF
 (THF OR THFS)
 122730 TETRAHYDROFURAN
 (TETRAHYDROFURAN OR THF)
 L3 51 L2 AND (SOLVENT OR HEXANE OR HEXENE OR HEPTANE OR HEPTENE OR
 TETRAHYDROFURAN)

 => s l3 and (dewax? (l) catalyst (l) solvent)
 5438 DEWAX?
 663991 CATALYST
 668562 CATALYSTS
 851063 CATALYST
 (CATALYST OR CATALYSTS)
 608403 SOLVENT
 302207 SOLVENTS
 766333 SOLVENT
 (SOLVENT OR SOLVENTS)
 180 DEWAX? (L) CATALYST (L) SOLVENT
 L4 2 L3 AND (DEWAX? (L) CATALYST (L) SOLVENT)

 => s l3 and oxidiz? (3a) catalyst
 373661 OXIDIZ?
 663991 CATALYST
 668562 CATALYSTS
 851063 CATALYST
 (CATALYST OR CATALYSTS)
 4429 OXIDIZ? (3A) CATALYST
 L5 1 L3 AND OXIDIZ? (3A) CATALYST

 => s l3 and (alkali or lithium or sodium or potassium or rubidium or cesium)
 380955 ALKALI
 4116 ALKALIS
 31228 ALKALIES
 400753 ALKALI
 (ALKALI OR ALKALIS OR ALKALIES)
 279687 LITHIUM
 352 LITHIUMS
 279811 LITHIUM
 (LITHIUM OR LITHIUMS)
 941549 SODIUM
 34 SODIUMS
 941558 SODIUM
 (SODIUM OR SODIUMS)
 539964 POTASSIUM
 15 POTASSIUMS
 539966 POTASSIUM
 (POTASSIUM OR POTASSIUMS)
 59829 RUBIDIUM
 4 RUBIDIUMS
 59830 RUBIDIUM
 (RUBIDIUM OR RUBIDIUMS)
 90258 CESIUM
 6 CESIUMS
 90258 CESIUM
 (CESIUM OR CESIUMS)
 L6 7 L3 AND (ALKALI OR LITHIUM OR SODIUM OR POTASSIUM OR RUBIDIUM OR
 CESIUM)

=> s l6 or l5 or l4

L7 10 L6 OR L5 OR L4

=> d l7 ibib ab 1-10

L7 ANSWER 1 OF 10 CAPLUS COPYRIGHT 2004 ACS on STN
ACCESSION NUMBER: 2003:590797 CAPLUS
DOCUMENT NUMBER: 139:119730
TITLE: Supported catalyst treatment
INVENTOR(S): Daage, Michel; Koveal, Russell John; Clark, Janet
Renee; Marler, David Owen
PATENT ASSIGNEE(S): USA
SOURCE: U.S. Pat. Appl. Publ., 13 pp.
CODEN: USXXCO
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2003144128	A1	20030731	US 2002-59928	20020129
WO 2003064040	A1	20030807	WO 2002-US41513	20021227
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, OM, PH, PL, PT, RO, RU, SD, SE, SG, SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UG, UZ, VN, YU, ZA, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG				

PRIORITY APPLN. INFO.: US 2002-59928 A 20020129
AB Supported metal catalysts for the hydrogenation of carbon monoxide to form a mixture of hydrocarbons are **regenerated** by decreasing the hydrocarbon content of the catalyst, preferably by contact with hydrogen-containing gas at elevated temps., impregnating under a non-oxidizing atmospheric with a solution of at least one of an ammonium salt and an alkyl ammonium salt, optionally in combination with up to five moles of ammonia per L of solution to the point where it has absorbed a volume of solution equal to at least .apprx.10% of its calculated pore volume; **oxidizing** the **catalyst** with a gaseous oxidant in the presence of the impregnating solution and **activating** the catalyst by reduction with hydrogen at elevated temps. Optionally, the catalyst is calcined after the oxidation step, and passivated after the **activation** step.

L7 ANSWER 2 OF 10 CAPLUS COPYRIGHT 2004 ACS on STN
ACCESSION NUMBER: 2002:814862 CAPLUS
DOCUMENT NUMBER: 137:312709
TITLE: Process for the preparation of promoted skeletal iron Fischer-Tropsch catalysts for hydrocarbon manufacture
INVENTOR(S): Zhou, Peizheng; Lu, Yijun
PATENT ASSIGNEE(S): Hydrocarbon Technologies, Inc., USA; Institute of Coal Chemistry
SOURCE: U.S. Pat. Appl. Publ., 7 pp., Cont.-in-part of U.S. Ser. No. 895,621.
CODEN: USXXCO
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 3
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
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US 2002156137	A1	20021024	US 2002-107915	20020327
US 6777452	B2	20040817		
US 6277895	B1	20010821	US 1999-399852	19990921
US 2002052423	A1	20020502	US 2001-895621	20010702
US 2003109591	A1	20030612	US 2002-267236	20021008
PRIORITY APPLN. INFO.:			US 1999-399852	A1 19990921
			US 2001-895621	A2 20010702
			US 2002-107915	A2 20020327

AB Promoted skeletal iron catalysts are described which contain 70-90% iron, 0-5.0% copper, 0.1-10.0% manganese, and 0.1-3.0% **potassium**, with the balance being aluminum. These catalysts are prepared by mixing the metal chips or powders uniformly together, then melting and rapidly quenching the molten metals to form a solid metal alloy precursor including the promotor metals except **potassium**, removing most of the aluminum by caustic (e.g., NaOH) extraction/leaching to provide a base skeletal iron form, then loading the **potassium** promoter from a suitable **potassium** alc. solution promoter (e.g., K₂CO₃ in MeOH). After evaporation of the **solvent**, the promoted skeletal iron catalyst is **activated** by contact with hydrogen. The promoted skeletal iron catalysts are utilized with synthesis gas (e.g., H₂-CO mixts.) for Fischer-Tropsch hydrocarbon manufacture at 10-30% catalyst concentration, 200-350°/1.0-3.0 MPa, and a gas-hourly space velocity of 0.5-5.0 h⁻¹ to produce hydrocarbon liquid products. The promoted skeletal iron catalysts provide good catalytic activity and selectivity for hydrogen and CO conversions, are attrition resistant, and are readily separable from the waxy liquid product by gravity sedimentation.

L7 ANSWER 3 OF 10 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1998:510502 CAPLUS

DOCUMENT NUMBER: 129:204988

TITLE: Lube oil processing--conclusion. Catalytic hydroprocessing a good alternative to **solvent** processing

AUTHOR(S): Helton, Terry E.; Degnan, Thomas F., Jr.; Mazzone, Dominick N.; McGuinness, Mary P.; Hilbert, Tim L.; Dougherty, Rick C.

CORPORATE SOURCE: Mobil Technology Co., Paulsboro, NJ, USA

SOURCE: Oil & Gas Journal (1998), 96(29), 58, 60-62, 64, 67
CODEN: OIGJAV; ISSN: 0030-1388

PUBLISHER: PennWell Publishing Co.

DOCUMENT TYPE: Journal

LANGUAGE: English

AB In order to reduce process costs and improve base-oil quality, Mobil Oil Corp. has developed several hydroprocessing **catalysts** as an alternative to conventional **solvent** processing: Mobil lube **dewaxing**, (MLDW), Mobil selective **dewaxing** (MSDW), and Mobil **wax** isomerization (MWI). This article is the last of three that cover Mobil's lubricating oil technologies. The first discussed the company's compositional monitoring approach to predict lubricating oil qualities; the second presented processes and com. examples. Cycle length no longer appears to be a concern for lubricating oil hydroprocessing and **dewaxing**. MLDW and isomerization **dewaxing catalysts** require very infrequent **regeneration**. These lengthened-**catalyst** cycles help make the catalytic hydroprocessing route preferable to **solvent** processing.

REFERENCE COUNT: 12 THERE ARE 12 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L7 ANSWER 4 OF 10 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1997:280554 CAPLUS

DOCUMENT NUMBER: 127:20589

TITLE: Activity and selectivity of precipitated iron

Fischer-Tropsch catalysts
AUTHOR(S): O'Brien, Robert J.; Xu, Liguang; Spicer, Robert L.;
Baoy, Shiqi; Milburn, Diane R.; Davis, Burtron H.
CORPORATE SOURCE: Center for Applied Energy Research, University of
Kentucky, 3572 Iron Works Pike, Lexington KY 40511,
USA
SOURCE: Catalysis Today (1997), 36(3), 325-334
CODEN: CATTEA; ISSN: 0920-5861
PUBLISHER: Elsevier
DOCUMENT TYPE: Journal
LANGUAGE: English

AB Low-temperature (230°C), slurry-phase Fischer-Tropsch synthesis (FTS) was conducted with precipitated iron-silicon **catalysts** under industrially relevant conditions (flow 3.1 L/h-g Fe, H₂-CO 0.7:1, and 1.31 MPa). The effects of **activation** gas (hydrogen, carbon monoxide, or syngas) and promoters (**potassium** and copper) on activity and selectivity were explored. Optimum **potassium** promotion was 4-5 atomic%, relative to iron. Promotion with copper lowered the reduction temperature and increased FTS activity, regardless of the **activation** gas used. Carbon monoxide **activation** gave the highest activity for a 100:4.4:5.2 (atomic%) Fe-Si-K **catalyst** while syngas **activation** was superior for a 100:4.4:2.6:5.2 Fe-Si-Cu-K **catalyst**. Selectivity of the FTS product was not affected by the **activation** gas employed or copper promotion; however, **potassium** promotion increased **wax** and alkene selectivity. A syngas **activated** 100:4.4:2.6:5.2 Fe-Si-Cu-K **catalyst** gave the best overall performance at 230°C. Alkene selectivity was >75% for the C₂-C₁₁ fraction, methane selectivity was <3 weight% and C₁₂ selectivity was >70 weight%.

REFERENCE COUNT: 29 THERE ARE 29 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L7 ANSWER 5 OF 10 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1980:588787 CAPLUS
DOCUMENT NUMBER: 93:188787
TITLE: New process dewaxes lube base stocks
AUTHOR(S): Smith, Kenneth W.; Starr, W. C.; Chen, N. Y.
CORPORATE SOURCE: Mobil Res. and Dev. Corp., Paulsboro, NJ, USA
SOURCE: Oil & Gas Journal (1980), 78(21), 75-8, 83-4
CODEN: OIGJAV; ISSN: 0030-1388
DOCUMENT TYPE: Journal
LANGUAGE: English

AB The Mobil Lube **Dewaxing** (MLDW) process **dewaxes** high-pour-point lubricant raffinates to low-pour-point high-quality lubricant base stocks by selective cracking of normal and slightly branched paraffins to lighter hydrocarbons which can be readily removed by distillation or stripping. The process uses 2 **catalysts**, a synthetic shape-selective zeolite to effect pour-point reduction and 1 to impart oxidation stability to the MLDW product. The process is a fixed-bed reaction system which operates at 250-3000 psig. The waxy charge is mixed with a H-rich gas, preheated to reaction temperature, and charged to the MLDW reactor system. The **dewaxed** product is flashed to sep, H-rich circulating gas. Excess product gas is removed from the system. The remaining hydrocarbons (boiling below the lubricant range) are removed by standard distillation or stripping, and the **catalyst** can be **reactivated**. The process operating parameters and yield and properties of the products obtained are discussed. The process offers the following advantages over **solvent dewaxing**: improvement of lubricant yields; achievement of the very low pour points (-40 to -60°F) required for transformer and refrigeration service; elimination of the need for a sep. product finishing operation; and reduction in operating costs. A range of products including circulating and industrial hydraulic oils, diesel and passenger car engine oils, gear oils, and transformer oil was formulated

with various MLDW base stocks.

L7 ANSWER 6 OF 10 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1974:426269 CAPLUS
DOCUMENT NUMBER: 81:26269
TITLE: Catalytic manufacture of modified polyamides
INVENTOR(S): Michel, Erich; Schwarz, Hans Hermann
PATENT ASSIGNEE(S): Mannesmann A.-G.
SOURCE: Ger. Offen., 9 pp.
CODEN: GWXXBX
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 2233087	A1	19740117	DE 1972-2233087	19720630
PRIORITY APPLN. INFO.:			DE 1972-2233087	19720630

AB Caprolactam(I) was melt-polymerized in the presence of silica gel and lubricants using Na caprolactamate (II) [2123-24-2] as **catalyst**, triisopropylaluminum [2397-67-3], triethylaluminum [97-93-8], or calcium hydride [7789-78-8] as **activator** to counteract the deactivating effect of the silica gel, and hexamethylene diisocyanate (III) [822-06-0] as accelerator to give polycaprolactam (IV) [25038-54-4] with modified phys. properties. Thus, a melt containing I, silica gel, synthetic **wax** (m.p. .sim.105.deg.) was mixed with (iso-Pr)₃Al and the nascent alc. evaporated in vacuo. The reaction mixture was mixed with II and III at .sim.125.deg. and heated 30 min at 150.deg. to give solid IV.

L7 ANSWER 7 OF 10 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1959:100033 CAPLUS
DOCUMENT NUMBER: 53:100033
ORIGINAL REFERENCE NO.: 53:18097f-i,18098a-i,18099a-c
TITLE: Steroids of natural configuration. I. Stereochemistry of lumisterol and 9 α -lumisterol (pyrocalciferol)
AUTHOR(S): Castells, J.; Jones, E. R. H.; Meakins, G. D.; William, R. W. J.
CORPORATE SOURCE: Univ. Manchester, UK
SOURCE: Journal of the Chemical Society, Abstracts (1959) 1159-68
CODEN: JCSAAZ; ISSN: 0590-9791
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

AB Lumisterol (I) and pyrocalciferol (II) were 2 of the 4 ergosterol stereoisomers involving variations in the configurations at the 9- and 10-positions. The preparation from these 2 sterols of pairs of C-9 epimers and comparison of their mol. rotations indicated that the accepted structures should be reversed, i.e., I has the 9 β -configuration and II the 9 α -configuration. This proposal was confirmed by a procedure involving elimination of the stereochem. differences at position 10. Degradation of I yielded a tricyclic ketone, de-A-lumist-22-en-5-one (III), identical with that derived from isopyrocalciferol (9 β -ergosterol) (IV). The use of the more systematic names 9 β -ergosterol and 9 α -lumisterol in place of isopyro- and pyrocalciferol was now desirable. 4,22-Ergostadien-3-one (V) (1.29 g.) in 250 cc. CHCl₃ cooled to -80° and 9 cc. 2% Br in CHCl₃ added dropwise, NEt₃ added, the **solvent** removed, and the residue dissolved in Et₂O gave 1.36 g. 22,23-dibromo-4-ergosten-3-one (VI), m. 195-8° (alc.), [α]_D 38° (c 0.9), λ 239 m μ , ϵ 15,600, ν 1681, 1620 cm.⁻¹ Debromination of VI with **activated** Zn dust gave a high yield of V, m. 127-31°, [α]_D 39° (c 1.1), λ 241 m μ , ϵ 16,800. VI (1.45 g.) in 15 cc. EtOAc treated 50 min. at -80° with O₃, N passed

in, the solution allowed to warm to room temperature, left 40 hrs. with 150 cc. AcOH, 50 cc. H₂O, and 6 cc. 26% H₂O₂, the oil refluxed with 2 g. Zn dust 1 hr., and separated gave after chromatography on silica gel 295 mg. 5-oxo-3,5-seco-A-nor-22-ergosten-3-oic acid (VII), m. 141-3°, $[\alpha]_D -4^\circ$ (c 1.0), ν 1706 and 966 cm.⁻¹ Following a similar reaction in which the debromination procedure was omitted gave 22,23-dibromo-5-oxo-3,5-seco-A-norergostan-3-oic acid, m. 196-9°, $[\alpha]_D 11^\circ$ (c 1.2). VII Na salt was prepared by titrating a solution of the acid in MeOH with MeOH-0.5N NaOMe. The salt (780 mg.) and 3.6 g. PhCH₂CO₂Na heated 3 hrs. at 295-300°/0.02-0.05 mm. gave 638 mg. distillate, chromatographed on Al₂O₃ to 404 mg. de-A-22-ergost-22-en-5-one, m. 101-3° (MeOH), $[\alpha]_D -30^\circ$ (c 1.4), ν 1715 cm.⁻¹ II (9.5 g.) in 200 cc. xylene and 100 cc. cyclohexanone dried by azeotropic distillation, refluxed 40 min. with 7 g. (iso-PrO)₃Al in xylene, 200 cc. AcOH and 500 cc. Tetralin added, the **solvents** removed at 140°/17 mm. and finally at 140°/0.01 mm., 50 cc. more Tetralin added, the **solvent** removed, the residue dissolved in dilute HCl and Et₂O, washed, and worked up gave an oil. This oil in 200 cc. CHCl₃ treated 45 min. at 20° with 4 cc. 50% HBr in AcOH and the product chromatographed on deactivated Al₂O₃ gave 3.56 g. 9 α -lumista-4,6,22-trien-3-one (VIII), m. 87-91° (MeOH), $[\alpha]_D 68^\circ$ (c 1.0), λ 288 m μ , ϵ 24,000, ν 1667, 1613, 1582, and 970 cm.⁻¹ VIII (3.32 g.) in 150 cc. alc. containing 15 mg. KOH hydrogenated over 750 mg. Pd-Norit until 1.03 mole H was absorbed and the crude product chromatographed gave 9 α -lumista-4,22-dien-3-one, m. 140-3° (alc.), $[\alpha]_D -53^\circ$ (c 0.9). The remainder of the crude product (3.15 g.) in 700 cc. CHCl₃ treated dropwise at -80° with 27.5 cc. 2% Br in CHCl₃, 0.5 cc. NEt₃ added, and the crude product chromatographed on silica gel gave 1.89 g. 22,23-dibromo-9 α -lumist-4-en-3-one (IX), m. 190-2° (alc.), $[\alpha]_D -37^\circ$ (c 1.2), λ 241 m μ , ϵ 15,000. X (1.79 g.) in 150 cc. EtOAc ozonized and treated 40 hrs. with AcOH and H₂O₂ and isolated gave 513 g. 5-oxo-3,5-seco-A-nor-9 α -lumist-22-en-3-oic acid (XI), m. 190-7° (alc.), $[\alpha]_D -43.5^\circ$ (c 1.1). XI (226 mg.) in 6 cc. dioxane treated dropwise with 9 cc. aqueous 0.061N Ba(OH)₂ and the residue heated at 300-50°/0.001 mm. gave after chromatography 19 mg. tricyclic ketone, m. 85.5-7.0° (MeOH), $[\alpha]_D 51.5^\circ$ (c 0.9), ν 1715, 967 cm.⁻¹ Lumista-4,7,22-trien-3-one (8.8 g.) in 700 cc. CHCl₃ treated 40 min. with 9 cc. 50% HBr and the crude product chromatographed on Al₂O₃ gave 1.7 g. unconjugated ketone, m. 177.5-80.5° (Me₂CO), λ 249 m μ , ϵ 21,300, ν 1712 cm.⁻¹ Elution with 2:1 ligroine-C₆H₆ gave 3.4 g. lumista-4,6,22-trien-3-one (XII), m. 99-101° (Me₂CO-MeOH), $[\alpha]_D -629^\circ$ (c 1.2), λ 287 m μ , ϵ 25,100, ν 1660, 1624, 1588, 973 cm.⁻¹ XII (4.8 g.) in 250 cc. alc. containing 22 mg. KOH hydrogenated over 760 mg. Pd-C, 1 drop AcOH and 1 drop C₅H₅N added, the **catalyst** removed, the **solvent** evaporated, and the residue chromatographed on Al₂O₃ gave 930 mg. putative 22-lumisten-3-one and 3.5 g. 4,22-lumistadien-3-one (XIII), m. 124-5.5° (alc.), $[\alpha]_D -170^\circ$ (c 1.05), λ 242 m μ , ϵ 16,700, ν 1675, 1623, 983 cm.⁻¹ XIII (4.01 g.) in 800 cc. CHCl₃ treated dropwise at -80° with 28 cc. 2% Br-CHCl₃, NEt₃ added, the **solvent** removed, the residue dissolved in Et₂O, and filtered gave 4.3 g. 22,23-dibromo-4-lumisten-3-one (XIV), m. 169-72° (Me₂CO-MeOH), $[\alpha]_D -102^\circ$ (c 0.8), λ 241 m μ , ϵ 15,900, ν 1681, 1618, cm.⁻¹ Debromination of XIV with **activated** Zn gave a good yield of XIII. XIV (3.95 g.) in 385 cc. EtOAc ozonized and treated with H₂O₂ as described above gave 3.78 g. 22,23-dibromo-5-oxo-3,5-seco-A-norlumistan-3-oic acid (XV), m. 177.5-9.0° (Me₂CO-ligroine), $[\alpha]_D 6^\circ$ (c 1.1), ν 1715 cm.⁻¹ XV (1.26 g.) refluxed 2 hrs. with 2.5 g. Zn dust in 1:1 Et₂O-MeOH, 1 g. more Zn dust added, and refluxing continued 1 hr. gave 800 mg. 5-oxo-3,5-seco-A-norlumist-22-en-3-oic acid (XVI), m. 78-80° (ligroine), $[\alpha]_D -11^\circ$ (c 0.9), ν 1712 and 967 cm.⁻¹; Me

ester, oil. Na salt of XVI (0.6 g.) heated 3.25 hrs. with 3 g. Na phenyl-acetate at 290-300°/0.05 mm. and the 435 mg. distillate chromatographed on Al2O3 gave 355 mg. III, m. 28-34°, [α]D -48° (c 0.96), ν 1715 and 968 cm.-1 III was wax-like and could not be crystallized by conventional methods but when pure it solidified spontaneously. III was recovered unchanged after treatment at room temperature with 0.5% p-MeC6H4SO3H in 1:1 AcOH-C6H6 or refluxed in MeOH

and

0.2N NaOMe; oxime m. 134.5-7.0°, [α]D -94° (c 0.7). IV (41 g.) in 900 cc. PhMe and 165 cc. cyclohexanone refluxed 5 hrs. with 25 g. (iso-PrO)3Al in 140 cc. dry distillate under N and the product chromatographed on Al2O3 gave 9.7 g. oil (A), 7.6 g. semicryst. material (B). A was rechromatographed to give 273 mg. 9β-ergosta-5,7,22-trien-3-one, m. 123-31° (MeOH), [α]D 521° (c 0.9), λ 272, 282, mμ, ε 9400 and 10,000, ν 1712 cm.-1, and 6.2 g. material which combined with B and crystallized gave 10.3 g. 9β-ergosta-4,7,22-trien-3-one (XVII), m. 110-12.5° (MeOH), [α]D 185° (c 1.3), λ 242 mμ, ε 14,200, ν 1667, 1626, 970 cm.-1 XVII (1.033 g.) in 40 cc. MeOH refluxed 45 min. with 2 cc. concentrated HCl and the material isolated by chromatography on

Al2O3

gave 549 mg. material, m. 105-17° (Me2CO-MeOH), λ 254 mμ, ε 19,300, ν 1712 cm.-1, and 280 mg. 9β-ergosta-4,6,22-trien-3-one (XVIII), m. 161-4° (Me2CO-MeOH), [α]D -14° (c 0.98), λ 284 mμ, ε 26,200, ν 1656, 1623, 1587, 965 cm.-1 XVIII (2.4 g.) in 370 cc. alc. containing 11 mg. KOH hydrogenated over 5% Pd-C and chromatographed gave 377 mg. putative 9β-ergost-22-en-3-one and 1.54 g. 9β-ergosta-4,22-dien-3-one (XIX), m. 155-7° (Me2CO-MeOH), [α]D 25° (c 1.0), λ 244 mμ, ε 14,500, ν 1667, 1616, 965 cm.-1 XIX (1.41 g.) in 300 cc. CHCl3 treated dropwise at -80° with 9.8 cc. Br in CHCl3, NEt3 added, the residue dissolved in Et2O, and filtered gave 1.4 g. 22,23-dibromo-9β-ergost-4-en-3-one (XX), m. 203.5-6.0° (Me2CO-alc.), [α]D 39° (c 0.9), λ 244 mμ, ε 16,700, ν 1669, 1621 cm.-1 Debromination of XX with Zn gave a high yield of XIX. XX (1.15 g.) in 300 cc. EtOAc ozonized as above and the acid isolated as before gave 760 mg. 22,23-dibromo-5-oxo-seco-A-nor-9β-ergostan-3-oic acid (XXI), m. 178-82° (Me2CO-ligroine), [α]D -9° (c 0.95), ν 1712 cm.-1 XXI (690 mg.) refluxed 2 hrs. with 1.3 g. Zn dust in 64 cc. 1:1 Et2O-MeOH, the process repeated, and the product isolated gave 5-oxo-3,5-seco-A-nor-9β-ergost-22-en-3-oic acid (XXII), m. 92-4°, [α]D -39° (c 1.0), ν 1712 cm.-1 XXII Na salt (395 mg.) and 1.2 g. PhCH2CO2Na heated 2.5 hrs. at 290-5°/0.02-0.05 mm. gave III.

L7 ANSWER 8 OF 10 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1957:69301 CAPLUS

DOCUMENT NUMBER: 51:69301

ORIGINAL REFERENCE NO.: 51:12547f-i

TITLE: Polyhydric alcohol esters of acids formed by oxidation of hydrocarbons

INVENTOR(S): Polly, Orville L.; Cunningham, Orin D.

PATENT ASSIGNEE(S): Union Oil Co.

DOCUMENT TYPE: Patent

LANGUAGE: Unavailable

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2783270		19570226	US	

AB Polymeric esters (I) of a mixture of acids produced by the controlled oxidation of paraffin wax (II) and glycols or alcs. containing 3-4 OH groups are oily liquids of low viscosity and plasticizers for vinyl

resins; the resulting resins have good low-temperature flexibility, mech. strength, and stability. Thus, air is introduced into an oxidation vessel containing refined II, m. 63°, at 130° and 100 lb./sq. in. at a rate of 5.5 cu. ft./bbl./min. until the acid number is 250 mg. KOH/g. and the saponification number is 490. The naphtha (III, b. 50-85°) insol. fraction (IV) of the oxidized **wax** has an acid number of 180 and saponification number of 330 and a O-carboxyl ratio of 1.5; the fraction soluble in III has an acid number of 160 and saponification number of 300. IV is saponified with excess caustic soda in the presence of alc., extracted with III, mixed with MeOH and concentrated H₂SO₄, and fractionated. One equivalent of the resulting Me ester (b₅ 90-260°, acid number 23, sapon number 241) is heated with 1-1.5 equivs. of ethylene glycol (V), diethylene glycol, 2-ethyl-**hexane** -1,3-diol, hexaethylene glycol, glycerol, or penta-erythritol at 205-50° in the presence of **alkali** hydroxides, carbonates, or alcoholates, e.g. (MeO)₂Mg, as **catalyst**. The properties of I prepared with V are: mol. weight 530, hydroxyl oxygen 1.2%, carbonyl oxygen 1.6%, acid number 1.4, saponification number 325, and Gardner color 13. I is also obtained by direct esterification of oxidized II and V in the presence of acid **catalysts** and purified with adsorbents, such as clay or **activated** charcoal.

L7 ANSWER 9 OF 10 CAPLUS COPYRIGHT 2004 ACS on STN
 ACCESSION NUMBER: 1947:13336 CAPLUS
 DOCUMENT NUMBER: 41:13336
 ORIGINAL REFERENCE NO.: 41:2701a-i,2702a-b
 TITLE: A new method of preparing substituted vinyl compounds. Depolymerization studies on vinyl polymers
 AUTHOR(S): Bachman, G. Bryant; Hellman, H.; Robinson, K. R.; Finholt, R. W.; Kahler, E. J.; Filar, L. J.; Heisey, L. V.; Lewis, L. L.; Micucci, D. D.
 CORPORATE SOURCE: Purdue Univ., Lafayette, IN
 SOURCE: Journal of Organic Chemistry (1947), 12, 108-21
 CODEN: JOCEAH; ISSN: 0022-3263
 DOCUMENT TYPE: Journal
 LANGUAGE: Unavailable

AB A new method has been developed for synthesizing substituted vinyl compds. by altering polymers chemically and then depolymerizing them with special application to halogenated styrenes and aromatic vinyl derivs. The various factors influencing depolymerization are studied. It is found that the chemical structure and the method of formation of the polymers have a great effect upon the yield of monomers and that polymers of higher mol. weight (>20,000) give higher yields of monomers than polymers of low mol. weight (<20,000). Rapid high-temperature heating of polymers seems to give better yields than slow heating at low temps. All **catalysts** tried, such as Al₂O₃, sand, NaOH, Fe filings, ceresin **wax**, or Silicone fluid, are without effect or decrease the yield. Recracking of partially cracked products gives no better yields. An O-free atmosphere in the cracking apparatus and addition of inhibitors to the receiving flask reduce the tendency of the distillate to polymerize. Of the sulfonated, nitrated, and chlorinated polystyrenes only the last can be depolymerized satisfactorily. Polystyrenes (I) of mol. wts. ranging from 3700-190,000 are prepared under conditions given in a table and are cracked, in addition to Dow Styron YA5K27 (II) (mol. weight 50,000) and Monsanto Lustron Molding Powder (III) (mol. weight 74,000), giving 30-65% monomers. I are chlorinated in CHCl₃, CCl₄, or (CHCl₂)₂, using various **catalysts**, to a Cl content of 36-45.7% and are isolated either by evaporating the **solvent** or by pouring the solution into EtOH. The distillates of several cracking expts. are fractionated, using a 75-cm. column and 2.5-5 mm. vacuum, giving 10% p-chlorostyrene, b_{2.5} 45.8-5.9°, n_{D20} 1.5648, d₂₅₂₅

1.090 (p-ClC₆H₄CO₂H m. 238-40°), 25% 3,4-dichlorostyrene, b₅ 81.6-3.5°, n_D28 1.5769 (3,4-Cl₂C₆H₃CO₂H m. 205-5.5°), and 15-20% unidentified volatile chlorinated products. The regenerated monomers polymerize readily in the presence of peroxide catalysts, giving rubbers which compare favorably with those obtained from Mathieson dichlorostyrene, but they copolymerize more slowly than the com. products. Bromination of 104 g. II in CCl₄ with 160 g. Br in the presence of FeCl₃ and cracking 100 g. at 20 mm. and 400° give 82.5 g. reddish distillate from which 25-35% monobromostyrene, b₁₃ 87°, d₂₅25 1.3891, n_D25 1.5850 (p-BrC₆H₄CO₂H m. 251-3°), is isolated. α-Methylstyrene (IV) is polymerized by dropwise addition of 535 g. to 600 cc. concentrated H₂SO₄ at -15° to -10° with vigorous stirring and treating the reaction product with steam. The poly-IV (V) (340 g.) is chlorinated in 1.5 l. CCl₄ in the presence of 1 g. FeCl₃ with 442 g. Cl at 0-5° with exclusion of light, giving 450 g. chlorinated V. Cracking of this product gives 6% of a mixture of Cl compds. from which 3,4-dichloro-IV (VI), b₉ 97-100°, n_D25 1.5746, d₂₅25 1.2675, is isolated. VI, b₃ 94°, n_D25 1.5732, d₂₅25 1.2242, is also prepared from Et 3,4-dichlorobenzoate (?; given as Et 3,4-dichlorobenzoic acid in original). VI is readily copolymerized to give excellent rubbers. Chlorination of 235 g. Cumar Resin W1 in 1.2 l. CCl₄ at 0-10° with exclusion of light in the presence of 1 g. FeCl₃ 6.5 hrs. with 390 g. Cl gives 417 g. chlorinated product (VII) containing slightly over 2.5 Cl atoms per indene unit. Cracking of 200 g. VII gives 53.4 g. distillate which is separated into 3 fractions, b₃₋₅ 75-260°, which are not investigated further. Nitration of 5 g. I (mol. weight 50,000) with 60 cc. fuming HNO₃ 1 hr. at 50° and pouring the reaction mixture into 1.25 l. H₂O give 6.5 g. nitrated product (VIII) containing 9.6-10.5% N. VIII decompose on heating without depolymerization. Reduction of 5 g. VIII in NH₄OH suspension with Na₂S₂O₄ gives 2.5 g. polyaminostyrene (IX). IX is readily diazotized and coupled with phenols to give dyes: 2-Cl₁₀H₇OH gives a red color, Neville and Winther acid a brown-red, Schaeffer's acid red, 1-Cl₁₀H₇OH brown, Broenner's acid brown, and PhNMe₂ red in acid, brown in neutral and basic solns. Sulfonation of II with 10 vols. concentrated H₂SO₄ at 100°, finally 8 hrs. at 160°, pouring the reaction mixture into ice-H₂O, neutralizing with NaOH, and evaporating to dryness give a mixture of Na₂SO₄ and Na polystyrenesulfonate which when heated with KCN chars. Attempts to alkylate I failed. In an attempt to acylate I with AcCl or BzCl in the presence of AlCl₃ it was found that I is rapidly degraded and rearranged by the AlCl₃ and upon cracking in the presence of 1-2% AlCl₃ a mixture is obtained which shows no unsatn. to Br and KMnO₄. Attempts to distill acetyl- or benzoylstyrene resulted in polymerization. The apparatus and the technique for the depolymerization are described.

L7 ANSWER 10 OF 10 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1944:534 CAPLUS
DOCUMENT NUMBER: 38:534
ORIGINAL REFERENCE NO.: 38:113b-h
TITLE: Catalytic hydrogenation of organic compounds
INVENTOR(S): Schmidt, Otto
PATENT ASSIGNEE(S): General Aniline & Film Corp.
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2322095		19430615	US	

AB This patent (as are also the patents of the next 4 higher patent nos.) is based on an application filed in the U. S. Patent Office April 1, 1931; corresponding patent protection are stated to have been applied for in Germany November 20, 1925. This patent relates generally to the production of alcs. by a process which involves heating a carboxylic organic

compound containing 2 or more C atoms in the liquid state in the presence of H and of a hydrogenating **catalyst**, which may include Cu, Co or Ni, together with an **activating** compound such as an O-containing compound of a metal of from the 1st to 7th groups of the Periodic System (suitably to a temperature of 120-300° under a pressure of 30-400 atmospheric); and more particularly relates to preparing alcs. by such a process from mixed carboxylic acids obtained by the destructive oxidation of paraffin **wax**. Mention is made of numerous initial materials and **catalysts** which may be used, and numerous examples are given with details of hydrogenation and statements of use of the alcs. obtained. U. S. 2,322,096 relates particularly to such production of alcs. from acids such as montanic acid or stearic acid or various acids containing 8-18 C atoms, with use of a **catalyst** such as Co **activated** by a hydroxide, oxide, carbonate, phosphate, silicate or nitrate of an **alkali**, alkaline earth or rare earth metal. U. S. 2,322,097 relates especially to the catalytic hydrogenation of anhydrides such as those of palmitic, stearic or lauric acid with use of a **catalyst** such as Co **activated** with K₂O. U. S. 2,322,098 relates particularly to hydrogenations of naphthenic acids at elevated temps. and pressures with a **catalyst** such as Co **activated** with vanadic acid or an **activating** agent as used in the process of U. S. 2,322,096. U. S. 2,322,099 relates to generally similar hydrogenations of esters such as methyl stearate, coconut oil, or castor oil for the production of octadecanediol, etc. Numerous other generally similar reactions are described or generally mentioned, and the various products obtained are of different consistencies and other properties such as to adapt them, variously, for use in the production of artificial **waxes** or cosmetic prepns., or washing, emulsifying or wetting agents by sulfonation, as softening agent for natural or synthetic rubber or rubberlike materials, and may be esterified, as by acetylation, to produce products suitable for use as assistant **solvents** in varnishes, lacquers or the like.

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<input type="checkbox"/>	L6	L5 and oxidiz\$3 near4 dewax\$3 near2 catalyst	0
<input type="checkbox"/>	L5	L4 and (alkali or lithium or sodium or potassium or rubidium or cesium)	17
<input type="checkbox"/>	L4	L3 and dewax\$3 near4 catalyst near5 solvent	28
<input type="checkbox"/>	L3	L2 and (solvent or hexane or hexene or heptane or heptene or tetrahydrofuran)	1169
<input type="checkbox"/>	L2	L1 and (regenerat\$3 or rejuvenat\$3 or activat\$3 or reactivat\$3)	1584
<input type="checkbox"/>	L1	wax with catalyst	4913

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☐ 1. Document ID: US 20030166451 A1

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L7: Entry 1 of 8

File: PGPB

Sep 4, 2003

PGPUB-DOCUMENT-NUMBER: 20030166451

PGPUB-FILING-TYPE: new

DOCUMENT-IDENTIFIER: US 20030166451 A1

TITLE: Fischer-tropsch catalyst enhancement

PUBLICATION-DATE: September 4, 2003

INVENTOR-INFORMATION:

NAME	CITY	STATE	COUNTRY	RULE-47
Koveal, Russell John	Baton Rouge	LA	US	
Daage, Michel	Baton Rouge	LA	US	
Shen, Eric Baochun	Baton Rouge	LA	US	

US-CL-CURRENT: [502/38](#); [518/715](#)

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	Claims	KWIC	Draw. De
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☐ 2. Document ID: US 20030144366 A1

L7: Entry 2 of 8

File: PGPB

Jul 31, 2003

PGPUB-DOCUMENT-NUMBER: 20030144366

PGPUB-FILING-TYPE: new

DOCUMENT-IDENTIFIER: US 20030144366 A1

TITLE: Catalyst regeneration

PUBLICATION-DATE: July 31, 2003

INVENTOR-INFORMATION:

NAME	CITY	STATE	COUNTRY	RULE-47
Daage, Michel	Baton Rouge	LA	US	
Koveal, Russell John	Baton Rouge	LA	US	
Chang, Min	McLean	VA	US	

APPL-NO: 10/ 059926 [\[PALM\]](#)

DATE FILED: January 29, 2002

INT-CL: [07] C07 C 27/00, C07 C 27/06, B01 J 38/64

US-CL-PUBLISHED: 518/715; 502/25

US-CL-CURRENT: 518/715; 502/25

ABSTRACT:

There is provided a process for regenerating the activity of used metal catalysts for the hydrogenation of carbon monoxide comprising decreasing the hydrocarbon content thereof, calcining under an oxidant-containing atmosphere, impregnating with a solution of at least one of a metal compound, calcining under an oxidant-containing atmosphere and activating by contacting with a hydrogen-containing gas at elevated temperatures to form an active catalyst. The process regenerates and enhances both supported and dispersed active metal (DAM) catalysts. Used catalysts enhanced by the process are initially treated to decrease their hydrocarbon content. The treatment may be carried out in a single reactor, or by carrying out up to all steps after catalyst may be withdrawn from a reactor and returned to at least one reactor, both preferably during operation thereof. Up to all steps may be effected in a subsequent reactor, or in specialized apparatus.

RELATED APPLICATIONS

[0001] The assignee of this application is filing herewith the following applications: Docket No. 37227, entitled "Fischer-Tropsch Catalyst Enhancement"; Docket No. 37228, entitled "Supported Catalyst Regeneration"; Docket No. 37229, entitled "Supported Catalyst Activation"; Docket No. 39158, entitled "Supported Catalyst Treatment"; and Docket No. 39773, entitled "Catalyst Enhancement". Also related in pending application Ser. No. 09/628,047, filed Aug. 1, 2000, entitled "process for Increasing Cobalt Catalyst Hydrogenation Activity Via Aqueous Low Temperature Oxidation".

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	Claims	KWIC	Draw De
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☐ 3. Document ID: US 20030144131 A1

L7: Entry 3 of 8

File: PGPB

Jul 31, 2003

PGPUB-DOCUMENT-NUMBER: 20030144131

PGPUB-FILING-TYPE: new

DOCUMENT-IDENTIFIER: US 20030144131 A1

TITLE: Catalyst enhancement

PUBLICATION-DATE: July 31, 2003

INVENTOR-INFORMATION:

NAME	CITY	STATE	COUNTRY	RULE-47
Koveal, Russell John	Baton Rouge	LA	US	
Daage, Michel	Baton Rouge	LA	US	
Mauldin, Charles Harrison	Baton Rouge	LA	US	
Clark, Janet Renee	Baton Rouge	LA	US	

APPL-NO: 10/ 059927 [PALM]
DATE FILED: January 29, 2002

INT-CL: [07] B01 J 20/34, B01 J 38/12

US-CL-PUBLISHED: 502/38
US-CL-CURRENT: 502/38

ABSTRACT:

A process for enhancing metal catalysts for carbon monoxide hydrogenation comprising reducing the catalyst so that at least a portion is in the metallic state, impregnating under a non-oxidative atmosphere with a solution of a salt of at least one metal, optionally in combination with at least one of an ammonium salt, an alkyl ammonium salt, a weak organic acid and ammonia, oxidizing with a gaseous oxidant in the presence of the impregnating solution, and reducing to form an active catalyst. Used catalysts enhanced by the process are initially treated to decrease their hydrocarbon content. The treatment may be carried out in a single reactor, or by carrying out up to all steps after catalyst may be withdrawn from a reactor and returned to at least one reactor, both preferably during operations. Up to all steps may be effected in a subsequent reactor, or in specialized apparatus.

RELATED APPLICATIONS

[0001] The assignee of this application is filing herewith the following applications: Docket No. 37227, entitled "Fischer-Tropsch Catalyst Enhancement"; Docket No. 37228, entitled "Supported Catalyst Regeneration"; Docket No. 37229, entitled "Supported Catalyst Activation"; Docket No. 39158, entitled "Supported Catalyst Treatment"; and Docket No. 39774, entitled "Catalyst Regeneration". Also related in pending application Ser. No. 09/628,047, filed Aug. 1, 2000, entitled "process for Increasing Cobalt Catalyst Hydrogenation Activity Via Aqueous Low Temperature Oxidation".

[0002] This invention relates to the production of higher hydrocarbons from synthesis gas utilizing a metal catalyst, particularly a cobalt catalyst.

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	Claims	KWIC	Draw. De
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☐ 4. Document ID: US 20030144129 A1

L7: Entry 4 of 8

File: PGPB

Jul 31, 2003

PGPUB-DOCUMENT-NUMBER: 20030144129
PGPUB-FILING-TYPE: new
DOCUMENT-IDENTIFIER: US 20030144129 A1

TITLE: Supported catalyst regeneration

PUBLICATION-DATE: July 31, 2003

INVENTOR-INFORMATION:

NAME

CITY

STATE

COUNTRY

RULE-47

Clark, Janet Renee	Baton Rouge	LA	US
Koveal, Russell John	Baton Rouge	LA	US
Daage, Michel	Baton Rouge	LA	US

APPL-NO: 10/ 059917 [PALM]

DATE FILED: January 29, 2002

INT-CL: [07] B01 J 23/94, B01 J 23/96

US-CL-PUBLISHED: 502/28; 502/38, 502/53

US-CL-CURRENT: 502/28; 502/38, 502/53

ABSTRACT:

There is provided a process for renewing the activity of used, supported metal catalysts for the hydrogenation of carbon monoxide to form a mixture of hydrocarbons comprising decreasing the hydrocarbon content of the catalyst, impregnating said catalyst under an non-oxidative atmosphere with a solution at least one weak organic acid, preferably a mono- or di-carboxylic acid, to the point where it has absorbed a volume of said solution equal to at least about 10% of its calculated pore volume, oxidizing the catalyst with a gaseous oxidant in the presence of the impregnating solution and activating the catalyst by reduction with hydrogen at elevated temperatures. Optionally, the catalyst is calcined after the oxidation step, and passivated after the activation step. A preferred means of decreasing the hydrocarbon content of the catalyst is contacting it with a hydrogen-containing gas at elevated temperatures.

RELATED APPLICATIONS

[0001] The assignee of this application is filing herewith the following applications: Docket No. 37227, entitled "Fischer-Tropsch Catalyst Enhancement" Docket No. 37229, entitled "Supported Catalyst Activation"; Docket No. 39158, entitled "Supported Catalyst Treatment"; Docket No. 39773, entitled "Catalyst Enhancement"; and Docket No. 39774, entitled "Catalyst Regeneration". Also related in pending application Ser. No. 09/628,047, filed Aug. 1, 2000, entitled "process for Increasing Cobalt Catalyst Hydrogenation Activity Via Aqueous Low Temperature Oxidation".

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	Claims	KWC	Draw. De
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☐ 5. Document ID: US 20030144128 A1

L7: Entry 5 of 8

File: PGPB

Jul 31, 2003

PGPUB-DOCUMENT-NUMBER: 20030144128

PGPUB-FILING-TYPE: new

DOCUMENT-IDENTIFIER: US 20030144128 A1

TITLE: Supported catalyst treatment

PUBLICATION-DATE: July 31, 2003

INVENTOR-INFORMATION:

NAME	CITY	STATE	COUNTRY	RULE-47
Daage, Michel	Baton Rouge	LA	US	
Koveal, Russell John	Baton Rouge	LA	US	
Clark, Janet Renee	Baton Rouge	LA	US	
Marler, David Owen	Baton Rouge	LA	US	

APPL-NO: 10/ 059928 [PALM]

DATE FILED: January 29, 2002

INT-CL: [07] B01 J 20/34, B01 J 38/70

US-CL-PUBLISHED: 502/23

US-CL-CURRENT: 502/23

ABSTRACT:

There is provided a process for renewing the activity of supported metal catalysts for the hydrogenation of carbon monoxide to form a mixture of hydrocarbons comprising decreasing the hydrocarbon content of the catalyst, preferably by contact with hydrogen-containing gas at elevated temperatures, impregnating under a non-oxidizing atmosphere with a solution of at least one of an ammonium salt and an alkyl ammonium salt, optionally in combination with up to five moles of ammonia per liter of solution to the point where it has absorbed a volume of solution equal to at least about 10% of its calculated pore volume; oxidizing the catalyst with a gaseous oxidant in the presence of the impregnating solution and activating the catalyst by reduction with hydrogen at elevated temperatures. Optionally, the catalyst is calcined after the oxidation step, and passivated after the activation step.

RELATED APPLICATIONS

[0001] The assignee of this application is filing herewith the following applications: Docket No. 37227, entitled "Fischer-Tropsch Catalyst Enhancement"; Docket No. 37228, entitled "Supported Catalyst Regeneration"; Docket No. 37229, entitled "Supported Catalyst Activation"; Docket No. 39773, entitled "Catalyst Enhancement"; and Docket No. 39774, entitled "Catalyst Regeneration". Also related in pending application Ser. No. 09/628,047, filed Aug. 1, 2000, entitled "process for Increasing Cobalt Catalyst Hydrogenation Activity Via Aqueous Low Temperature Oxidation".

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	Claims	KWIC	Draw. De
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☐ 6. Document ID: US 6777451 B2

L7: Entry 6 of 8

File: USPT

Aug 17, 2004

US-PAT-NO: 6777451

DOCUMENT-IDENTIFIER: US 6777451 B2

TITLE: Catalyst enhancement

DATE-ISSUED: August 17, 2004

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Koveal; Russell John	Baton Rouge	LA		
Daage; Michel	Baton Rouge	LA		
Mauldin; Charles Harrison	Baton Rouge	LA		
Clark; Janet Renee	Baton Rouge	LA		

ASSIGNEE-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY	TYPE	CODE
ExxonMobil Research and Engineering Company	Annandale NJ				02	

APPL-NO: 10/ 059927 [PALM]

DATE FILED: January 29, 2002

PARENT-CASE:

RELATED APPLICATIONS The assignee of this application is filing herewith the following applications: Ser. No. 10/059,916, entitled "Fischer-Tropsch Catalyst Enhancement"; Ser. No. 10/059,917, entitled "Supported Catalyst Regeneration"; Ser. No. 10/059,918, entitled "Supported Catalyst Activation"; Ser. No. 10/059,928, entitled "Supported Catalyst Treatment"; and Ser. No. 10/059,926, entitled "Catalyst Regeneration", all filed Jan. 29, 2002. Also related in pending application Ser. No. 09/628,047, filed Aug. 1, 2000, entitled "process for Increasing Cobalt Catalyst Hydrogenation Activity Via Aqueous Low Temperature Oxidation". This invention relates to the production of higher hydrocarbons from synthesis gas utilizing a metal catalyst, particularly a cobalt catalyst.

INT-CL: [07] C07 C 27/00, B01 J 20/34, B01 J 23/40, B01 J 23/72

US-CL-ISSUED: 518/710; 518/700, 518/709, 518/713, 518/715, 502/38, 502/53, 502/54, 502/326, 502/331

US-CL-CURRENT: 518/710; 502/326, 502/331, 502/38, 502/53, 502/54, 518/700, 518/709, 518/713, 518/715

FIELD-OF-SEARCH: 518/700, 518/709, 518/710, 518/713, 518/715, 502/38, 502/53, 502/54, 502/326, 502/331

PRIOR-ART-DISCLOSED:

U.S. PATENT DOCUMENTS

PAT-NO	ISSUE-DATE	PATENTEE-NAME	US-CL
<u>2369956</u>	February 1945	Feisst et al.	260/449.6
<u>2542558</u>	February 1951	Naragon et al.	252/416
<u>3256205</u>	June 1966	Constabaris et al.	252/413
<u>3661798</u>	May 1972	Cosyns et al.	252/416
<u>3839191</u>	October 1974	Johnson	208/108
<u>4191664</u>	March 1980	McArthur	252/466
<u>4399234</u>	August 1983	Beuther et al.	518/715
<u>4795726</u>	January 1989	Schaper et al.	502/26
<u>4814066</u>	March 1989	Fu	208/120
<u>4888131</u>	December 1989	Goetsch et al.	252/373

<u>4929336</u>	May 1990	Lowery et al.	208/120
<u>4954244</u>	September 1990	Fu et al.	208/120
<u>4978689</u>	December 1990	Bell et al.	518/709
<u>5160456</u>	November 1992	Lahn et al.	252/373
<u>5283216</u>	February 1994	Mitchell	502/30
<u>5292705</u>	March 1994	Mitchell	502/325
<u>5389592</u>	February 1995	Weissman et al.	502/25
<u>5438028</u>	August 1995	Weissman et al.	502/202
<u>5495055</u>	February 1996	Rueter	568/881
<u>5728918</u>	March 1998	Nay et al.	585/733
<u>6130184</u>	October 2000	Geerlings et al.	502/350
<u>6201030</u>	March 2001	Beer	518/709
<u>6331574</u>	December 2001	Lapidus et al.	518/709
<u>6465529</u>	October 2002	Daage et al.	
<u>2001/0047041</u>	November 2001	Lapidus et al.	

FOREIGN PATENT DOCUMENTS

FOREIGN-PAT-NO	PUBN-DATE	COUNTRY	US-CL
4302992	August 1994	DE	
0244014	November 1987	EP	
0244014	November 1987	EP	
0583837	February 1994	EP	
0583837	February 1994	EP	
0979673	February 2000	EP	

OTHER PUBLICATIONS

A. Khodakov et al., "Structural Modification of Cobalt Catalysts: Effect of Wetting Studied by X-Ray and Infrared Techniques", Oil & Gas Science and Technology, Rev. IFP, vol. 54 (1999), No. 4. pp. 525-535.

ART-UNIT: 1621

PRIMARY-EXAMINER: Parsa; J.

ATTY-AGENT-FIRM: Simon; Jay Marin; Mark D.

ABSTRACT:

A process for enhancing metal catalysts for carbon monoxide hydrogenation comprising reducing the catalyst so that at least a portion is in the metallic state, impregnating under a non-oxidative atmosphere with a solution of a salt of at least one metal, optionally in combination with at least one of an ammonium salt, an alkyl ammonium salt, a weak organic acid and ammonia, oxidizing with a gaseous oxidant in the presence of the impregnating solution, and reducing to form an active catalyst. Used catalysts enhanced by the process are initially treated to decrease their hydrocarbon content. The treatment may be carried out in a single reactor, or by carrying out up to all steps after catalyst may be withdrawn from a reactor and returned to at least one reactor, both preferably during operations. Up to all steps may be effected in a subsequent reactor, or in specialized apparatus.

39 Claims, 0 Drawing figures

Full	Title	Citation	Front	Review	Classification	Date	Reference	Claims	KWIC	Draw D
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☐ 7. Document ID: US 6753354 B2

L7: Entry 7 of 8

File: USPT

Jun 22, 2004

US-PAT-NO: 6753354

DOCUMENT-IDENTIFIER: US 6753354 B2

TITLE: Fischer-tropsch catalyst enhancement

DATE-ISSUED: June 22, 2004

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Koveal; Russell John	Baton Rouge	LA		
Daage; Michel	Baton Rouge	LA		
Shen; Eric Baochun	Baton Rouge	LA		

ASSIGNEE-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY	TYPE	CODE
ExxonMobil Research and Engineering Company	Annandale	NJ			02	

APPL-NO: 10/ 059916 [PALM]

DATE FILED: January 29, 2002

INT-CL: [07] C07 C 27/00, B01 J 20/34, B01 J 23/40, B01 J 23/72

US-CL-ISSUED: 518/715; 518/700, 518/709, 518/710, 518/713, 502/38, 502/53, 502/54, 502/326, 502/331

US-CL-CURRENT: 518/715; 502/326, 502/331, 502/38, 502/53, 502/54, 518/700, 518/709, 518/710, 518/713

FIELD-OF-SEARCH: 518/700, 518/709, 518/710, 518/713, 518/715, 502/38, 502/53, 502/54, 502/326, 502/331

PRIOR-ART-DISCLOSED:

U.S. PATENT DOCUMENTS

PAT-NO	ISSUE-DATE	PATENTEE-NAME	US-CL
<u>2369956</u>	February 1945	Feisst et al.	260/449.6
<u>2542558</u>	February 1951	Naragon et al.	252/416
<u>3256205</u>	June 1966	Constabaris et al.	252/413
<u>3661798</u>	May 1972	Cosyns et al.	252/416
<u>3839191</u>	October 1974	Johnson	208/108
<u>4172053</u>	October 1979	Vogt et al.	252/447

<u>4191664</u>	March 1980	McArthur	252/466
<u>4399234</u>	August 1983	Beuther et al.	518/715
<u>4795726</u>	January 1989	Schaper et al.	502/26
<u>4814066</u>	March 1989	Fu	208/120
<u>4888131</u>	December 1989	Goetsch et al.	252/373
<u>4929336</u>	May 1990	Lowery et al.	208/120
<u>4954244</u>	September 1990	Fu et al.	208/120
<u>4978689</u>	December 1990	Bell et al.	518/709
<u>5070058</u>	December 1991	Sawicki et al.	502/206
<u>5160456</u>	November 1992	Lahn et al.	252/373
<u>5283216</u>	February 1994	Mitchell	502/30
<u>5292705</u>	March 1994	Mitchell	502/325
<u>5389592</u>	February 1995	Weissman et al.	502/25
<u>5438028</u>	August 1995	Weissman et al.	502/202
<u>5495055</u>	February 1996	Rueter	568/881
<u>5728918</u>	March 1998	Nay et al.	585/733
<u>5863856</u>	January 1999	Mauldin	502/325
<u>6130184</u>	October 2000	Geerlings et al.	502/350
<u>6201030</u>	March 2001	Beer	518/709
<u>6331574</u>	December 2001	Lapidus et al.	518/709
<u>6355593</u>	March 2002	Daage et al.	502/111

FOREIGN PATENT DOCUMENTS

FOREIGN-PAT-NO	PUBN-DATE	COUNTRY	US-CL
4302992	August 1994	DE	
0244014	November 1987	EP	
0244014	November 1987	EP	
0583837	February 1994	EP	
0583837	February 1994	EP	
0979673	February 2000	EP	

OTHER PUBLICATIONS

A. Khodakov et al., "Structural Modification of Cobalt Catalysts: Effect of Wetting Studied by X-Ray and Infrared Techniques", Oil & Gas Science and Technology, Rev. IFP, vol. 54 (1999), No. 4. pp. 525-535.

ART-UNIT: 1621

PRIMARY-EXAMINER: Parsa; J.

ATTY-AGENT-FIRM: Simon; Jay Marin; Mark D.

ABSTRACT:

There is provided a process for hydrocarbon synthesis wherein a supported metal catalyst for hydrogenating carbon monoxide to form a mixture of hydrocarbons is regenerated by decreasing its hydrocarbon content, impregnating under a non-oxidative atmosphere with a solution of at least one member selected from the group

consisting of ammonium salts, alkyl ammonium salts and weak organic acids, optionally including ammonia, oxidizing with a gaseous oxidant in the presence of the impregnating solution, activating the catalyst by reduction with hydrogen at elevated temperatures and reusing the catalyst. The treatment may be carried out in a single reactor, or by carrying out up to all steps after catalyst has been withdrawn therefrom and returned to at least one reactor. Up to all steps subsequent to decreasing the hydrocarbon content may be effected in a subsequent reactor, or in specialized apparatus.

55 Claims, 0 Drawing figures

Full	Title	Citation	Front	Review	Classification	Date	Reference		Claims	KMC	Draw D
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☐ 8. Document ID: US 6753286 B2

L7: Entry 8 of 8

File: USPT

Jun 22, 2004

US-PAT-NO: 6753286

DOCUMENT-IDENTIFIER: US 6753286 B2

TITLE: Supported catalyst regeneration

DATE-ISSUED: June 22, 2004

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Clark; Janet Renee	Baton Rouge	LA		
Koveal; Russell John	Baton Rouge	LA		
Daage; Michel	Baton Rouge	LA		

ASSIGNEE-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY	TYPE	CODE
ExxonMobil Research and Engineering Company	Annandale	NJ			02	

APPL-NO: 10/ 059917 [PALM]

DATE FILED: January 29, 2002

PARENT-CASE:

RELATED APPLICATIONS The assignee of this application is filing herewith the following applications: Ser. No. 10/059,916, entitled "Fischer-Tropsch Catalyst Enhancement"; Ser. No. 10/059,918, entitled "Supported Catalyst Activation"; Ser. No. 10/059,928, entitled "Supported Catalyst Treatment"; Ser. No. 10/059,927, entitled "Catalyst Enhancement"; and Ser. No. 10/059,926, entitled "Catalyst Regeneration", all filed Jan. 29, 2002. Also related in pending application Ser. No. 09/628,047, filed Aug. 1, 2000, entitled "process for Increasing Cobalt Catalyst Hydrogenation Activity Via Aqueous Low Temperature Oxidation".

INT-CL: [07] B01 J 38/62, B01 J 20/34, B01 J 38/12

US-CL-ISSUED: 502/28; 502/38, 502/53

US-CL-CURRENT: 502/28; 502/38, 502/53

FIELD-OF-SEARCH: 502/28, 502/38, 502/53

PRIOR-ART-DISCLOSED:

U.S. PATENT DOCUMENTS

PAT-NO	ISSUE-DATE	PATENTEE-NAME	US-CL
<u>2369956</u>	February 1945	Feisst et al.	260/449.6
<u>2542558</u>	February 1951	Naragon et al.	252/416
<u>3256205</u>	June 1966	Constabaris et al.	252/413
<u>3661798</u>	May 1972	Cosyns et al.	252/416
<u>3839191</u>	October 1974	Johnson	208/108
<u>4191664</u>	March 1980	McArthur	252/466
<u>4399234</u>	August 1983	Beuther et al.	518/715
<u>4454240</u>	June 1984	Ganguli	502/26
<u>4522928</u>	June 1985	McVicker et al.	502/26
<u>4795726</u>	January 1989	Schaper et al.	502/26
<u>4814066</u>	March 1989	Fu	208/120
<u>4888131</u>	December 1989	Goetsch et al.	252/373
<u>4929336</u>	May 1990	Lowery et al.	208/120
<u>4954244</u>	September 1990	Fu et al.	208/120
<u>4978689</u>	December 1990	Bell et al.	518/709
<u>5160456</u>	November 1992	Lahn et al.	252/373
<u>5283216</u>	February 1994	Mitchell	502/30
<u>5292705</u>	March 1994	Mitchell	502/325
<u>5356845</u>	October 1994	Clavenna et al.	502/21
<u>5389592</u>	February 1995	Weissman et al.	502/25
<u>5438028</u>	August 1995	Weissman et al.	502/202
<u>5495055</u>	February 1996	Rueter	568/881
<u>5728918</u>	March 1998	Nay et al.	585/733
<u>6201030</u>	March 2001	Beer	518/709
<u>6239054</u>	May 2001	Shukis et al.	502/29
<u>6331574</u>	December 2001	Lapdius et al.	518/709

FOREIGN PATENT DOCUMENTS

FOREIGN-PAT-NO	PUBN-DATE	COUNTRY	US-CL
4302992	August 1994	DE	
19963409	July 2001	DE	
0244014	November 1987	EP	
0244014	November 1987	EP	
0583837	February 1994	EP	
0583837	February 1994	EP	
0979673	February 2000	EP	

OTHER PUBLICATIONS

A. Khodakov et al., "Structural Modification of Cobalt Catalyst: Effect of Wetting Studied by X-Ray and Infrared Techniques", Oil & Gas Science and Technology, Rev.

IFP, vol. 54 (1999), No. 4. pp. 525-535.

ART-UNIT: 1754

PRIMARY-EXAMINER: Silverman; Stanley S.

ASSISTANT-EXAMINER: Strickland; Jonas N.

ATTY-AGENT-FIRM: Simon; Jay Marin; Mark D.

ABSTRACT:

There is provided a process for renewing the activity of used, supported metal catalysts for the hydrogenation of carbon monoxide to form a mixture of hydrocarbons comprising decreasing the hydrocarbon content of the catalyst, impregnating said catalyst under an non-oxidative atmosphere with a solution at least one weak organic acid, preferably a mono- or di-carboxylic acid, to the point where it has absorbed a volume of said solution equal to at least about 10% of its calculated pore volume, oxidizing the catalyst with a gaseous oxidant in the presence of the impregnating solution and activating the catalyst by reduction with hydrogen at elevated temperatures. Optionally, the catalyst is calcined after the oxidation step, and passivated after the activation step. A preferred means of decreasing the hydrocarbon content of the catalyst is contacting it with a hydrogen-containing gas at elevated temperatures.

24 Claims, 0 Drawing figures

Full	Title	Citation	Front	Review	Classification	Date	Reference	Claims	KMIC	Draw De
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Term	Documents
CATALYST	798365
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OXIDIZ\$3	0
OXIDIZ	4631
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OXIDIZABE	5
OXIDIZABI	10
OXIDIZABL	102
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